MASTER

Towards absolute non-simulation-based HR-EBSD on single experimental patterns, by means of excess-deficiency correction

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Summary

For material studies or characterisation where the individual effects of the different grains, phases or even grain boundaries are important, sub-grain 'micro- to nano-scale' spatial resolution absolute stress/strain measurement techniques are required. Examples of such studies include: furthering the understanding of grain boundary deformation mechanics, measuring of residual stresses and the design, study and characterisation of high performance metals.

Over the last two decades, the possibility of using the relatively simple and high spatial resolution Scanning Electron Microscope (SEM) technique of Electron Backscatter Diffraction (EBSD) to accomplish this goal has been explored. Even though EBSD is frequently used for crystal phase identification and crystal orientation mapping, it holds much more data and can be used to determine elastic stress/strain through measuring of the shift in zone axis caused by this elastic stress/strain. This started with correlating multiple differently strained EBSD patterns together allowing to measure this relative shift and thus relative elastic stress/strain, which is frequently referred to as high-Angular-Resolution EBSD (HR-EBSD). Eventually, by using dynamically simulated patterns as a strain-free reference even absolute stresses/strains could be measured. However, this came with some significant limitations, which may turn out to be permanent show-stoppers, namely that for all current simulation-based HR-EBSD techniques, the EBSD pattern center (PC) and the stress/strain state can not be determine simultaneously. As a result, one of these, usually the PC, has to be determined pre-correlation, with typical deviation in this determination already causing strain errors of $\sim 10^{-3}$, typically referred to as "phantom strains", effectively negating the actual strain accuracy of this technique.

Recently another absolute HR-EBSD was proposed by Vermeij *et al.* [1], which does not rely on simulated patterns. Instead, through co-correlating the pattern centers of multiple EBSD patterns from different grains, fully exploiting crystal symmetry and plane-stress inside an Integrated Digital Image Correlation (IDIC) framework, the PC, detector distance (DD), crystal orientation and the absolute stress/strain state can be simultaneously determined. Consequently, this technique is not limited by these "phantom strains", moreover, the strain accuracy was better than 10^{-4}, which has never been achieved, or even attempted, in HR-EBSD literature. However, this was only achieved on dynamically simulated patterns in a virtual case-study. Some factors observed in experimental EBSD patterns would deteriorate this strain accuracy. Further validation of this framework on experimental patterns was thus required.

This work takes a first step towards achieving accurate experimental stress/strain measurements using a non-simulation-based HR-EBSD method. Initially experimental factors of error are minimized by adapting the framework as to only require a single pattern, creating the first single-pattern non-simulation-based HR-EBSD method. On dynamically simulated patterns, a strain accuracy below 10^{-4} is also observed. Although, for experimental patterns strain errors nearly 50 times greater are observed. Through a strain error and residual analysis involving both dynamically simulated patterns, with different levels of realism, and experimental patterns, the excess-deficiency (E/D) effect, which is responsible for strong asymmetry of Kikuchi bands, is determined to be the main cause of experimental strain errors.

This work reports a first integrated correction of the E/D effect for any HR-EBSD technique, which is based on the iterative optimization of the scaling and blurring of the gradient in y of the pattern as to approximate and subsequently correct for the E/D effect. This is combined with an investigation and subsequent exclusion, based on possible future changes to the experimental setup, of regions with very large E/D effects. Altogether, this results in a final strain accuracy below $1.1 \cdot 10^{-3}$ reported for direct-electron-detector strain-free single-crystal silicon samples. It must be noted that, currently, either σ_{11} or σ_{22} has to be assumed and locked to gain convergence. Finally, there is much more room for optimization of the methodology to decrease the strain error and allow simultaneous correlation of the full in-plane stress tensor (σ_{11}, σ_{22} and σ_{12}) e.g. by pre-determining the DD, more advanced multi-pattern single-grain correlations, adjustments to the experimental setup and better approximating the experimental E/D effect.